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## Search History

**DATE: Monday, October 20, 2003** Printable Copy Create Case

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| L9 | L8 and 149.clas. | 16 | L9 |
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| L7 | black adj powder | 3360 | L7 |
| L6 | L5 not l2 | 12 | L6 |
| L5 | L4 and 149.clas. | 12 | L5 |
| L4 | perchlorate same organic same crystal | 83 | L4 |
| L3 | perchlorate same (organic near crystal) | 0 | L3 |
| L2 | L1 and 149.clas. | 5 | L2 |
| L1 | phenolphthalein | 4115 | L1 |

END OF SEARCH HISTORY

# WEST Search History

DATE: Monday, October 20, 2003

| Set Name side by side | Query | Hit Count | Set Name result set |
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| | *DB=USPT,PGPB; PLUR=YES; OP=OR* | | |
| L16 | L15 and microns | 22 | L16 |
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| L11 | potassium adj nitrate | 7849 | L11 |
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| L7 | black adj powder | 3360 | L7 |
| L6 | L5 not l2 | 12 | L6 |
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| L1 | phenolphthalein | 4115 | L1 |

END OF SEARCH HISTORY

L16: Entry 3 of 22 File: USPT Jan 7, 2003

DOCUMENT-IDENTIFIER: US 6503350 B2
TITLE: Variable burn-rate propellant

Brief Summary Text (16):
In accordance with one aspect of the present invention there is provided a mixed solid propellant. The propellant comprises a first propellant composition comprising a substantially homogeneous mixture of fuel particles distributed throughout a matrix of a first oxidizer, and a second propellant composition comprising a fuel and a second oxidizer. In preferred embodiments, the second propellant is present in a quantity sufficient to modify the burn rate of the first propellant to achieve a preselected burn rate and/or the fuel particles and first oxidizer are present in stoichiometric quantities. The fuel particles are preferably micron or nanometer-scale particles, preferably metals. In especially preferred embodiments, the fuel particles are aluminum and the oxidizer is ammonium perchlorate.

Brief Summary Text (41):
The lower atomic number fuels are desirable in that they have the potential to lower the weight of the motor relative to that for aluminum-based motors. One possible key to the success of such fuels is the existence of an appropriate passivation layer around the metallic particle. That passivation layer exists with aluminum in the form of Al.sub.2 O.sub.3. The Al.sub.2 O.sub.3 layer maintains the stability of the energetic aluminum particle while it is in intimate contact with the ammonium perchlorate oxidizer. If the reaction kinetics are too slow for these fuels when micron-sized particles are used, then nanometer-scale powders can be utilized.

Brief Summary Text (42):
The metallic particles of one preferred embodiment may be prepared by methods known in the art. Micron-sized metallic particles may be formed by methods involving mechanical comminution, e.g., milling, grinding, crushing. Such micron sized particles are commercially available from several sources, including Valimet of Stockton, Calif., and are relatively inexpensive.

Brief Summary Text (43):
Because the burn rate for a mixture of metallic fuel particles and oxidizer particles is dependent in part on average particle size, if a faster burn rate is desired, for some embodiments of the present invention it may be advantageous to use particles smaller than micron sized metallic particles produced by mechanical comminution. Nanometer-scale particles may be prepared by either the gas condensation method or the ALEX (exploded aluminum) method. In the gas condensation method, aluminum metal is heated to a vapor. The vapor then collects and condenses into particles. The particles thus produced are nominally spherical, approximately 40 nm in diameter and have a very tight size distribution (.+-.5 nm to 10 nm). These particles are single crystals with negligible structural defect density and are surrounded by an aluminum oxide passivation layer approximately 2.5 nanometers in thickness.

Brief Summary Text (45):
The rate of energy release for conventional metal fuels is relatively slow because of the relatively large (micron-sized) particle sizes utilized. Nanometer-sized metal powders demonstrate superior performance in this regard by virtue of their very small particle size. Because of the particles' very small size, both the thermal capacity of each particle and the distance from the core of the particle to the outer surface area where chemical reactions can take place are greatly reduced. Preferably, the metal fuel particles used in preferred embodiments of compositions and propellants have a diameter of about 10 nanometers to about 40 micrometers, more preferably about 10 nanometers to about 10 microns. In one preferred embodiment, the fuel particles have a diameter of about 0.1 micrometer to 1 micrometer. In other preferred embodiments, the fuel

particles have a diameter of about 20 nanometers to about 40 nanometers. Methods of preparing nanometal particles are known in the art (e.g. "Oxidation Behavior of Aluminum Nanoparticles", C. E. Aumann, G. L. Skofronick, and J. A. Martin, J. Vac. Sci. Technol. B 13(3), 1178, (1995); "Ultrafine Metal Particles", C. G. Granqvist and R. A. Buhrman, J. Appl. Phys., 47, 2200, (1976).).

Brief Summary Text (48):
There are several other preferred oxidizers for use in accordance with one preferred embodiment, including hydroxy ammonium perchlorate (HAP), ammonium nitrate (AN), cyclotetramethylene tetranitramine (HMX), cyclotrimethylene trinitramine (RDX), triaminoguanidine nitrate (TAGN), lithium perchlorate, sodium perchlorate, potassium perchlorate, lithium nitrate, sodium nitrate, and potassium nitrate. Any of these or other oxidizers, or mixtures thereof, may be used in preferred embodiments provided that they are capable of being dissolved, dispersed, suspended, emulsified or otherwise distributed into suitably small portions when placed in a solvent or solvent system such as a mixed solvent or emulsion, which may be polar, nonpolar, organic, aqueous, or some combination thereof. Preferred solvents or solvent systems are selected on the basis of their ability to dissolve, solvate, or disperse the oxidizer, while maintaining a minimum of reactivity towards the metallic fuel and oxidizer, at least for the time needed to complete the reaction. In accordance with a preferred embodiment, water is used as the solvent for AP.

Brief Summary Text (51):
The weight ratio of AP to aluminum for a stoichiometric mixture, i.e., no excess oxidizer or fuel, is 42:19. AP will generally not react with aluminum oxide (Al.sub.2 O.sub.3), favoring reaction with unoxidized aluminum metal, so the passivation layer forming the surface of the aluminum particle must be taken into consideration when calculating the proportions of AP to Al for a more precise stoichiometric mixture. When the aluminum is in the form of micron-sized particles, the Al.sub.2 O.sub.3 passivation layer, which is approximately 2.5 nm thick, is practically negligible in weight compared to that of the unoxidized metallic aluminum within the particle. However, when the aluminum is in the form of nanometer-sized particles, the aluminum oxide passivation layer can comprise a substantial portion of the total weight of the particle, e.g., 30 to 40 wt. % or more. Therefore, when nanometer-sized particles are used, less oxidizer per unit weight aluminum fuel is needed for a stoichiometric mixture.

Brief Summary Text (53):
Minimizing the reactant diffusion distance using conventional methods of preparing propellants can be difficult. If the metallic fuel particles and oxidizer particles are mechanically mixed into a powder, then in order to minimize reactant diffusion distance, the metallic particles and oxidizer particles should both be as small as possible. Under the current state of the art, nanometer scale metal particles can be prepared. However, the smallest particle sizes that have commonly been achieved for ammonium perchlorate are on the order of a few microns in diameter. Therefore, if nanometer metal particles are used with micron-sized (e.g., 3 .mu.m in diameter) oxidizer particles, reducing the particle size of the metal further will not have an appreciable effect on reactant diffusion distance since the oxidizer particle diameter dominates.

Brief Summary Text (69):
Once the solution is prepared and the solid particles are generally uniformly dispersed in solution, it is rapidly cooled to freeze the solution and fix the spatial distribution of particles throughout the solution. Any suitable cooling and freezing method may be used, but preferred methods involve immersing the solution in a cryogenic liquid, e.g., liquid nitrogen. The frozen liquid is then transferred to a vacuum chamber where solvent is removed by sublimation. This method works well with nanoaluminum since the metal is sufficiently non-reactive at cryogenic temperatures. In addition, the method is particularly well suited for use with nanoaluminum since nanometer-sized particles remain suspended in the solvent for a period of time than do micrometer-sized particles. This feature enables the nanoaluminum mixture to be rapidly frozen without undue settling of the aluminum particles to the bottom of the freezing volume, with little or no agitation required during freezing. Nanometer-sized particles form a pseudo-colloidal suspension with the solvent, whereas micron-sized particles rapidly settle out of the mixture unless continuous agitation is applied during freezing.

Detailed Description Text (21):
Means for reducing the binder content include increasing the particle size of the AP

component to as much as 200 microns, thus decreasing the surface area to be wetted by the binder. While the standard particle size of AP is 30 microns, it ranges from 3 to 200 microns in various formulations. However, this increased particle size may result in a corresponding undesirable decrease in power or burn rate, as discussed elsewhere herein. Therefore, a means of decreasing binder content without increasing AP component particle size is desirable.

Detailed Description Text (41):
Macroparticles of powder comprising particles of fuel/oxidizer matrix can be prepared by pressing or compacting the loose powder into pellets. Other suitable methods for consolidating the particles may also be used, e.g., thermal or chemical sintering. The pellets are then broken up into appropriately-sized macroparticles. Preferred macroparticles may be on the order of a few microns to several hundred microns in diameter. For example, macroparticles may be made which are approximately 30 microns or 200 microns, which are approximate sizes of commonly-used metal fuel and oxidizer particles in conventional solid rocket propellant formulations. The formation of macroparticles aids in mixing the NRC-4 with propellant components having a larger particle size than the NRC-4, because homogeneity is more easily approximated in a mixture of similarly sized particles than in one with particles of differing sizes. As such, in accordance with one embodiment of the present invention, there is provided a propellant comprising macroparticles and a binder/oxidizer mixture, wherein the macroparticles are an agglomeration of smaller particles of a composition comprising a substantially homogeneous mixture of fuel particles distributed throughout a matrix of an oxidizer.

Detailed Description Text (44):
Macroparticles of NRC-4 powder were prepared by compressing the powder into solid, flat pellets using a laboratory press. The pellets thus produced were ground into smaller pieces using a mortar and pestle. Macroparticles ranging in diameter from 100 microns to 250 microns were separated out by sifting the macroparticles through two sieves atop each other. The first sieve had 250 micron openings and the second sieve had 100 micron openings.

Detailed Description Text (50):
Table 4 presents the results of tests on two propellant formulations of the present invention using NRC-4 powder. The amount of AP listed in the composition is the stoichiometric amount of AP for the HTPB present, that is the amount of AP needed to react the HTPB only. The NRC-4, as discussed supra includes AP in a quantity sufficient to react with all the aluminum component thereof. Table 5 presents the results of tests on three more conventional propellant formulations in which the components as listed are micron-sized and are mixed together and cast into the tubes without curing. The AP listed in the formulations of Table 5 is the stoichiometric amount for both the Al and HTPB present. The formulations in Table 5 do not comprise the intimate, homogeneous mixtures of aluminum and AP of the compositions of the present invention, including NRC-4. All compositions in both tables, however, have about 12% HTPB. All percentages herein are by weight.

Detailed Description Text (53):
Comparison of the data in Table 4 to formulation 5 in Table 5 shows that the Propulsion Potential is increased about 8-fold when the fuel and its oxidizer is in the form of an intimate, substantially homogeneous mixture of nanoaluminum and AP according to a preferred embodiment (NRC-4) of the present invention. In these formulations, the NRC-4 provides small fuel particle size, on the order of about 40 nm, as well as low reaction diffusion distance because the nanoaluminum is dispersed throughout the AP oxidizer phase in a substantially uniform fashion. In preferred embodiments of fuel/oxidizer matrix compositions, such as NRC-4 and similar compositions comprising larger, micron-size fuel particles, the concerns regarding obtaining a homogeneous mixture of fuel and oxidizer seen in formulation 3 are minimized, because the composition itself, having the fuel particles dispersed throughout the oxidizer phase provide a mixture which is substantially homogeneous, intimate, and of the correct stoichiometry.

Detailed Description Text (85):
Therefore, by choosing the proper size metal fuel particles to include in a composition according to preferred embodiments of the present invention in which the fuel particles are distributed substantially uniformly throughout a stoichiometric amount of oxidizer, a propellant could be made having a preselected burn rate. For example, if a propellant were desired which had a burn rate slower than NRC-4, one could prepare a propellant according to the methods described above for NRC-4 in which the nanoaluminum is replaced with a larger sized particle, of a size up to and including particles several

microns in diameter. A micron-fuel based propellant would be advantageous in that micron sized aluminum is commercially available and is cheaper per pound than is nanoaluminum as of this date. Furthermore, adjustment of the burn rate by increasing the particle size allows for the adjustment without adding a low burn rate component, such as HTPB, which provides little power per pound. Thus, basing a propellant on a composition according to the present invention based upon micron-sized fuel particles could provide a propellant well suited for use in applications such as the Space Shuttle, Delta rockets, or other commercial aerospace vehicles, for which nanoaluminum based propellants such as NRC-4, which if used without a low burn rate material, may prove more energetic than is necessary.

Current US Class (1):
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